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APPLICATIONS OF
X-RAY DIFFRACTION AND RADIOGRAPHY IN
ENGINEERING AND RESEARCH



X-ray Laboratory
State Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Ga.
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The x-ray laboratory at the Georgia Tech Engineering Experiment Station was created for the primary purpose of rendering x-ray services in connection with engineering and research problems. This paper has been prepared in the belief that it is not only the function of the x-ray laboratory and its personnel to offer x-ray services but also to point out possible ways in which these services might be used to advantage.

Most of us are familiar with some form of radiography and, by only a slight stretch of our imaginations, can perhaps divine the majority of its possible applications to our particular problems. Fewer of us, however, are sufficiently familiar with x-ray diffraction so that we can predict its possible applications to a problem at hand. This paper lists numerous specific applications in the hope that an awareness of some of the possibilities of x-ray diffraction and radiography will be useful to the engineer and to the research worker.

This laboratory now has facilities to offer almost all of the applications listed, as well as many that are not listed, and the facilities are being expanded systematically.

The personnel of the x-ray laboratory are always happy to discuss any possible x-ray applications or techniques with all interested persons.

X-RAY DIFFRACTION

Because of the ease with which information concerning the submicroscopic structure of any sort of material may be obtained by x-ray diffraction--information usually obtainable only by inference from other methods of examination--this method of crystal analysis has become indispensable to modern industry. The great number of industrial applications already made prove the value of these fine structure studies, and provide a firm practical foundation for this branch of science. Hence the chemist, the physicist, the metallurgist, and the engineer now have in x-ray diffraction a powerful scientific instrument for use in the quest for better methods and improved materials, and in the maintenance of desired quality throughout the manufacturing processes.

Following is an outline of some of the many actual practical applications of x-ray crystal analysis that are being made. It will serve to indicate the universality of x-ray diffraction methods and to give an idea of the uses which may be made of this valuable method of analysis. Only those applications are included which are completely accepted by recognized authorities and which can be demonstrated beyond doubt as being useful to the engineer or research worker. A detailed discussion of each use is not given here, but each application is covered by one or more authoritative papers which have appeared in the physical, chemical, or engineering literature. It should be noted that most of the uses listed can be and actually are being used in many laboratories as routine testing methods.

APPLICATIONS TO METALLURGY AND METALLOGRAPHY

1. The Composition and Structure of Alloys.

(a) Identification of alloy components and compounds.

This is a special case of the general problem of chemical analysis by

x-ray diffraction, and is used routinely in many laboratories as a check on the results of other methods of examination.

(b) Differentiation between compound formation and solid solution. This is also a special case of chemical analysis in which a compound formed between two or more elements will give rise to a new x-ray pattern which is different from that of any of the constituents, while the solid solution will in general give the pattern of one of the elements, but with a shift in line positions which depends upon the relative amounts of the other elements present in the solution.

(c) Routine determination of percentage composition of solid solution phases, on the basis of measurement of line shift with varying amounts of solute present.

(d) Determination of the mechanism of alloy formation. This involves study of reflection and back reflection patterns of a series of alloys with various thermal treatments, and the correlation of the conclusions with chemical and microscopic data.

(e) Determination of miscibility limits and solid-phase boundaries of many-component alloy systems, by correlating lattice parameters with increasing percentage of alloying constituents.

(f) Working out and checking the details of the solidus phases of the equilibrium diagrams of binary and many-component alloy systems. X-ray diffraction analysis is the most convenient and dependable of the accepted modern methods for this purpose.

(g) The most rational classification of alloy types and systems has been made on the basis of x-ray crystal analysis.

(h) Study of the "order-disorder" phenomena in alloy systems.

2. The Effects of Rolling and Working on Metals and Alloys.

- (a) Determination of structural changes accompanying successive reductions of sheet and wire, as a comparison of methods of reduction by different techniques.
- (b) Study of the effect of initial grain size, carbon content, initial strip thickness, and of rolling variables on the final structure of rolled strip steel in determining the proper scientific methods of working and forming.
- (c) Determination of the effects of twisting and bending strip and wire.
- (d) Measurement of the extent of deformation and distortion by rolling, drawing, shaping, etc., as a routine check on the efficacy of the manufacturing process.
- (e) Determination of slip planes, "fiber" structure, etc., of rolled sheet and drawn wires.
- (f) Differentiation between surface and interior structures, or study of "zonal" structural characteristics.
- (g) Determination of the most desirable structure of a sheet or wire to be subjected to a forming operation, and a rational method of classifying metals as to workability. This method is used in many laboratories to "grade" every production lot. In this way the sheet mill can guarantee delivery of metal best suited to the manufacturer's shaping processes.
- (h) Furnishes an explanation of structural failures in spinning, cupping, and stamping operations. "Trouble shooting" in regard to these operations is one of the best paying uses of x-ray crystal analysis in the metallographic laboratory.
- (i) Measurement of the depth of cold work caused by machining, drilling, punching, grinding, etc.

(j) Study of the mechanism of "fatigue" and other types of metal failures, and in many cases a determination of the cause for premature or unexpected failures.

3. The Effects of Annealing and Other Thermal Treatments on Metals.

(a) Establishment and routine maintenance of scientifically correct annealing techniques, and in many cases also for heat treating techniques.

(b) Study of recrystallization mechanism, and exact determination of recrystallization temperature.

(c) Study of precipitation and age hardening phenomena.

(d) Study of the relation of carbon content to annealing, and the relations between amount of reduction, time and temperature of anneal, and the final structure.

(e) Determination of quench and temper structures of spring steels, and a continuous check on hardening and tempering operations.

(f) Study of growth of texture in castings.

(g) Measurement of strain relief upon annealing.

(h) Determination of surface effects, such as decarburization, oxidation, excessive crystal growth, etc., as differentiated from interior structure.

4. Miscellaneous Applications to Metals.

(a) Determination of true "crystal size" as distinguished from microscopic "grain size". This is a common and much used procedure in many factories.

(b) Determination of the structure of welds and the presence of strain or distortion in the neighborhood of the weld.

(c) Determination of the reason for and indication of the cure for "embrittlement" of malleable iron.

(d) Measurement of crystal size, crystal orientation, and absence of distortion (or degree of crystal perfection) in relation to electrical and magnetic properties of transformer steels.

(e) Determination of the effects of thermal treatments on the "spoilage" and recovery of permanent magnet alloys.

(f) Determination of uniformity, depth, and mechanism of surface hardening.

(g) Measurement of crystal size, preferred orientation, and thickness of electrodeposited films, a routine check on the plating process.

(h) Determination of the chemical composition of protective films, and study of mirrors and sputtered films.

(i) Study of the effects of included and absorbed or adsorbed gases on the structure of metals.

(j) Determination of optimum crystal size and best structure for electrical contact points, and a continuous check on these during manufacture.

(k) Study of the effects of crystal size and crystal orientation on electrical properties.

(l) Aid in the study of corrosion and corrosion or thermal "fatigue" and chemical embrittlement, and determination of the chemical composition of boiler scales.

(m) Furnishes a scientific approach to the preparation of new alloys, and a prediction of the properties of new or untried alloys.

(n) Study of the transition zone between base and covering of plated or enameled metals.

(o) Rational determination of the effects of minute impurities upon the structure of metals.

(p) Identification of inclusions in metals. This is a special case of chemical analysis by x-ray diffraction.

(q) An absolute and non-destructive measure of residual elastic surface stresses in metals. This is used quite extensively in several countries in the study of steel structures such as bridges and building frameworks.

(r) Determination of particle size in the colloidal region.

APPLICATIONS IN CHEMISTRY

1. General and Physical Chemistry

(a) Determination of ultimate crystal structure, including lattice types, unit cell dimensions, atomic positions, ionic groupings, and crystallographic systems of substances.

(b) Furnishes a unique and unquestionable characterization of individual chemical compounds. This is the basis of the wide-spread use of x-ray diffraction for chemical analysis. The analysis is, of course, made in terms of chemical compounds rather than in terms of elements and ionic groupings.

(c) Differentiation between a mixture, solid solution or complex compound formation.

(d) Supplies a quantitative estimate of the relative amounts of the various compounds in a mixture. The estimate can be refined by the proper use of a recording microphotometer.

(e) Furnishes a certain test for the crystallinity or non-crystallinity of a material, either in the solid state or in solution.

(f) Determination of crystal sizes in the microscopic and sub-microscopic (colloidal) ranges.

(g) Study of allotropic modifications and transitions of an element or compound, and the effects of impurities on these.

(h) Determination of the ideal or theoretical density of a substance, giving a basis for the estimation of porosity.

- (i) Differentiation between true and false hydrates.
(Chemical analysis.)
- (j) Discovery of unsuspected chemical reactions.
- (k) Recognition of colloiddally dispersed phases, and differentiation between true solutions and suspensions.
- (l) Determination of crystal size and structure of colloidal sols and gels.
- (m) Identification of adsorbed films and chemical changes involved in adsorption.
- (n) Determination of optimum crystal sizes and orientations for maximum catalytic activity, and study of the mechanism of catalysis and "poisoning" of catalysts. This is used not only to find the best processes for preparing a catalyst but also as a routine test of production.
- (o) Determination of molecular sizes in liquid solutions, and molecular weights of liquids.
- (p) Determination of the mechanism and course of dry reactions and allotropic transformations in the solid state, even at extremely high or extremely low temperatures.

2. Organic Chemistry

The list given above for General and Physical Chemistry, and in addition furnishes:

- (a) A sure test for the identity or non-identity of synthetic and naturally occurring materials.
- (b) Estimation of molecular weights of hydrocarbons, etc.
- (c) Measurement of atomic sizes, interatomic distance and diameters of molecules.
- (d) A method of following chemical reactions, as for example addition to or oxidation of a multiple bond.

- (e) Estimation of the purity of soaps, acids, etc.
- (f) Estimation of the positions of side chains and functional groups.
- (g) Measurement of the thickness of oriented films.
- (h) Determination of molecular orientation in fibers, and molecular structure of naturally occurring fibers and membranes.
- (i) A method of following polymerization and condensation reactions, and decomposition in breaking up long chain compounds.
- (j) Study of lubrication and lubricants, including a routine method of quantitatively comparing efficiencies of lubricants.
- (k) Study of changes taking place in the ripening of cheese, and during other processing of dairy products.
- (l) A rational classification of synthetic and natural plastics, and a qualitative scheme for identification of these.

3. Analytical Chemistry.

In addition to the applications listed above, x-ray diffraction provides for:

- (a) Identification of the chemical composition of precipitates.
- (b) Tests for purity and identification of impurities in precipitates.
- (c) Measurement of particle (crystal) sizes of precipitates in relation to treatment and reagent concentrations.
- (d) Determination of the state of perfection of the crystal lattice in precipitates, particularly in regard to aging effects, etc.

APPLICATIONS IN THE PROCESS INDUSTRIES

Since the process industries are engaged in chemical manufacture, the general applications listed under "chemistry" could be repeated here. To avoid duplication, however, only those applications of x-ray crystal analysis to some particular problems will be given.

1. Paints and Pigments.

(a) Structure and crystal sizes as functions of color, spreading, wetting and obscuring power, stability, gloss, and method of preparation.

(b) Study of the drying and setting of oils, the mechanisms of the reactions involved, etc., and their relationships to the structure and composition of pigments.

(c) Tests for solution of driers, and study of the mechanisms of their action.

(d) Routine analysis for purity of pigments. This is an important production test, particularly for those pigments which can exist in more than one crystal form, as for example titanium dioxide.

2. Ceramics and Glass.

(a) Routine qualitative and quantitative analysis of materials and clay mixtures, in terms of compounds present.

(b) Determination of the structural and chemical changes occurring during sintering, fusing, and other thermal treatments and the mechanisms of these reactions.

(c) Furnishes the best and fastest method for determining and checking the solidus phases of many component systems, and for determining miscibility limits.

(d) Gives a definite test for incipient devitrification of glass.

(e) Identification of substances imparting color or opacity to glasses or enamels.

(f) Determination of crystal size with relation to color of pigment.

(g) Study of transition zones between base metal and vitreous enamel.

(h) Measurement of chemical reaction rates in melt or during sintering.

3. Cement and Plaster.

(a) Study of reaction rates and mechanisms taking place during manufacture and use of cement.

(b) Routine chemical analysis of raw materials and clinker.

(c) Differentiation between particle size of aggregates and true crystal size.

(d) Method of determining and checking complex phase diagrams with certainty.

(e) Investigation of setting accelerators and their effects on the final structure of concrete.

(f) Control analysis of lime for crystal size, etc., to ensure proper plastic properties of plaster.

(g) Study of structure of limestone and its kiln behavior in relation to the properties of the final product.

(h) Study of the dehydration of gypsum and the structural changes involved in the use and re-use of plaster of paris molds.

4. Storage Batteries.

(a) Study of physical and chemical structure of plates as related to performance.

(b) Study of chemical reactions occurring during charge and discharge.

(c) Study of the influence of the structure of grid and composition and aging of the paste upon the physical properties of the plates, and control analysis for the manufacturing process.

(d) Identification of deposits and sediments on plates, separators, and in cell.

5. Rubber and Allied Products.

(a) Study of chemical reactions taking place during vulcanization and other processing.

(b) Determination of crystallinity, state of dispersions, crystal sizes of fillers, etc., and their relation to the physical characteristics of the finished products.

(c) Study of the basic structure of rubber and rubber-like materials. X-ray diffraction furnishes the only sure test of the fundamental relationships between natural and synthetic rubber.

(d) Study of fabrics and other binding materials used in the manufacture of rubber products, and routine grading of fibers as explained below.

6. Textiles and Fibers.

(a) Determination of the degree of "fibering". A quantitative relationship between the degree of fibering and tensile strength of cotton fibers has been developed and is being used as a routine method of grading cotton.

(b) Furnishes a scientific method of classifying cotton, silk, wool, and other natural and synthetic fibers.

(c) Determination of the rate, mechanism, and completeness of mercerization, nitration, and other chemical reactions, and use in control analysis.

(d) Determination of the mechanism of fire-proofing fibers, and of exact amount of reagent required.

(e) Identification of adsorbed films and the chemical changes involved in adsorption, particularly as applied to dyeing of fibers.

(f) Great improvements in quality, tensile strength, and non-wrinkling properties of rayon and other synthetic fibers has been made through x-ray studies. The development of artificial wool from

skim milk, peanuts, beans, etc., can be traced directly to x-ray diffraction studies of the structures of the various proteins. The development of "nylon", the new synthetic silk, has depended to a great degree on x-ray studies of its fiber characteristics by x-ray diffraction.

(g) X-ray diffraction studies on collagen fibers (side walls of animal intestines, tendons, etc.) have resulted in enormous improvement in the quality and wearing properties of tennis racket strings, and in the strength and controlled digestibility of surgical ligatures and sutures.

APPLICATIONS IN MINERALOGY

1. General Mineralogy.

(a) Complete and unambiguous mineralogical analysis of ores, clays, and other mineral mixtures.

(b) Analysis of industrial dusts, and correlation with the occurrence of industrial diseases.

(c) Classification and evaluation of certain commercial ores.

(d) Identification and classification of the clay minerals and complexes making up the so-called soil-colloid.

(e) A scientific method of studying the changes produced in natural minerals by weathering, accelerated weathering tests, and other chemical and physical degradations.

(f) Specifications for asbestos, mica, and other natural insulating materials for special purposes.

(g) Classification of coal, charcoal, etc.

2. Precious Stones and Gems.

(a) Identification, classification, and differentiation of genuine, both natural and synthetic, and imitation gems by a non-destructive test.

(b) Differentiation between natural and synthetic gems, non-destructively.

(c) Differentiation between natural and cultured pearls, non-destructively. This is a routine procedure with some of the leading jewelry manufacturers throughout the world.

(d) Determination of the proper orientation for a "jeweled" bearing (in watches, electric meters, etc.) to give maximum service and wearing qualities.

(e) Selection and classification of "black" diamonds for drills and dies, determination of causes for undue wear, and proper crystallographic orientations for optimum service.

(f) Determination of the proper direction of cutting quartz crystals for crystal oscillators in radio broadcasting and telephone equipment.

APPLICATIONS IN PHYSIOLOGY, PATHOLOGY, AND BIOLOGY

1. The applications under this heading are quite recent developments and are not yet generally used. Listing of some, however, will serve to show the general trend and possibilities of x-ray diffraction research in these complex and difficult, but extremely important fields.

(a) Differentiation between some normal and pathological tissues.

(b) Study of the effects of diseases on the structures of tissues, as on bone structure changes in rickets, cancer of the bone, and other bone diseases.

(c) Study of structure of living tissue, as nerve and muscle, in relation to body functions.

(d) Identification and classification of mineral deposits in organs, such as calcifications, gall stones, siliceous deposits, etc. Much interest is evident at present in the study of the action of free

quartz on lung tissue in silicosis, and of other industrial diseases and their occurrence, and many papers have been published in medical journals on x-ray diffraction studies of silicotic lung tissue.

(e) Structure and classification of tooth enamel, dentyne, etc., and structures of the teeth in relation to diet.

2. Papers of interest to pharmacists have appeared recently on the following subjects:

- (a) Identification of minerals in rhubarb.
- (b) Differentiation between natural and synthetic camphor.
- (c) Study of the reactions between menthol and the mercuric oxides.

RADIOGRAPHY

As stated at the outset, most of us are familiar with some form of radiography, at least medical radiography. Less familiar are the multitude of industrial applications. Objects for investigation by this non-destructive testing method may range in size from huge flumes or entire bridges to microtomic sections or flowers. Voltages vary from several million to a few thousand.

The bulk of industrial radiographic inspections employ voltages between 30 kv and 440 kv. The x-ray laboratory has facilities for radiography from the very lowest voltages up to 150 kv. This voltage range is suitable for most of the applications listed below, including microradiography. The maximum thickness that can be successfully radiographed with this equipment is about 25 gm/cm². This is equivalent to about 1½ inches of steel, or 3½ inches of aluminum. The following listing is intended to be indicative only, and not by any means complete.

Typical radiographic investigations

1. Castings and forgings may be investigated for

- (a) inclusions
- (b) porosity

- (c) cracks

2. Welds may be investigated for

- (a) fusion and penetration
- (b) inclusions, porosity, and cracks
- (c) undercutting (burn-through)
- (d) crater cracks (shrinkage cracks)

3. Concealed assemblies may be investigated for

- (a) orientation and position of parts differing in density from the surrounding medium (e.g., metal in plastic, rubber, plaster, etc.)
- (b) location of channeling in objects such as engine blocks, water-cooled dies, walls, concrete construction, etc.
- (c) inspection of packages, overstuffed furniture, persons, auto tires, etc. for concealed contraband.
- (d) assembly line inspection for proper filling of containers.

4. Microradiography

This is a relatively new radiographic technique which is capable of yielding three-dimensional information about samples less than 1/100 inch thick. Radiographs of very small specimens are taken with grainless film. The film is then enlarged to a usable size, thus giving an effective magnification similar to optical micrography. The technique has been of particular use in metallurgy.

5. Diffusion studies

This type of study is usually made by adding heavy atoms to the liquid whose diffusion rate is to be studied. Some examples of such studies are

- (a) diffusion of preservative liquids into hams and other food stuffs.
- (b) in biology and medicine, location of organs or regions where certain solutions tend to concentrate.
- (c) diffusion along the veins of a great variety of veined materials, including leaves and flowers.

6. Low-voltage radiography is used for such things as

- (a) radiography of paintings and documents to establish authenticity or reveal alterations.
- (b) investigation of paper products. This technique shows the grain of the paper.
- (c) investigation of glued joints for adhesion.